

≡ JP 2003 171325

(3)

<p>2004-046543/05 A41 E14 SUMO 2001.11.30 SUMITOMO CHEM CO LTD *JP 2003171325-A 2001.11.30 2001-366246(+2001JP-366246) C07C 37/11, 39/15 // C07B 61/00 Manufacture of solid organic compound e.g. 3,3',5,5'-tetra-alkyl biphenyl-2,2'-diol, involves oxidizing raw material organic compound by adding hydrogen peroxide to aqueous medium, below liquid level of aqueous medium C2004-019171</p>	<p>A(1-E13) E(10-E2A, 11-Q1)</p>
<p><u>NOVELTY</u> A raw material organic compound is oxidized by adding hydrogen peroxide to an aqueous medium containing the raw material organic compound, to obtain a solid organic compound. The hydrogen peroxide is added below the liquid level of the aqueous medium.</p> <p><u>USE</u> For producing solid organic compound, such as 3,3',5,5'-tetra-alkyl biphenyl-2,2'-diol.</p> <p><u>ADVANTAGE</u> The manufacturing method provides solid organic compounds with a high yield rate, by suppressing the decomposition of hydrogen</p>	<p>peroxide. The method uses biphenols which easily float on the liquid level of the aqueous medium during oxidation.</p> <p><u>DESCRIPTION OF DRAWING</u> The figure shows the schematic drawing of a reaction tank. (Drawing includes non-English language text). Stirring shaft 2 Reaction tank 3 Double pipe 4 Inner pipe 5 Outer pipe 6</p> <p><u>EXAMPLE</u> Water (in weight parts) (322) and lauric acid (3), were stirred at 85 ° C. Subsequently, caustic soda (209) was added, followed by the addition of 2,4-di-t-butylphenol (299). The contents were stirred for a while. 35% hydrogen peroxide (70) was added slowly at preset liquid depth, such that the temperature was maintained at 80-90 ° C. The contents were stirred at that temperature for 30 minutes. 35 %</p> <p>JP 2003171325-A+</p>

hydrogen peroxide (9) was further added and stirred. The contents were cooled to 65 ° C, and sodium sulfite (37) was added. After decomposition of surplus hydrogen peroxide, 98 % sulfuric acid (71) was added. Xylene (725) was then added, and the reaction product was dissolved. After dissolution, stirring was stopped and aqueous layer was separated. The obtained xylene solution was concentrated at 120 ° C under reduced pressure. A residue containing 3,3',5,5'-tetra-*t*-butyl biphenyl-2,2'-diol, was obtained at a yield of 78 %.

#### TECHNOLOGY FOCUS

Organic Chemistry - Preferred Compounds: The solid organic compound is a compound which floats on the liquid level of the aqueous medium. The raw material organic compound is a phenol of formula (II).

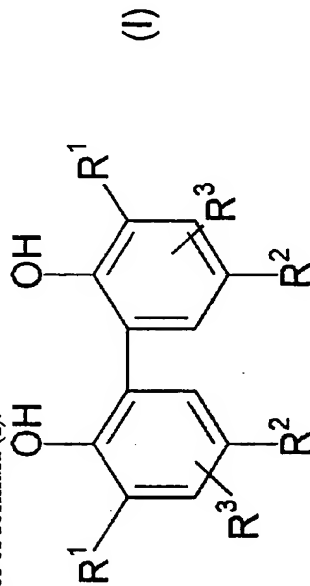
R<sup>1</sup> = cycloalkyl substituted by hydrogen atom, unsubstituted alkyl, alkyl substituted by phenyl, unsubstituted phenyl, phenyl substituted by alkyl, unsubstituted cycloalkyl, or alkyl;

R<sup>2</sup> = cycloalkyl substituted by unsubstituted alkyl, alkyl substituted by phenyl, unsubstituted phenyl, phenyl substituted by alkyl, unsubstituted cycloalkyl, or alkyl; and

R<sup>3</sup> = H or unsubstituted alkyl.

The phenyl in the alkyl group which is substituted by phenyl of R<sup>1</sup> and

R<sup>2</sup>, is further substituted by alkyl. The solid organic compound is a bisphenol of formula (I).



R<sup>1</sup>, R<sup>3</sup> = as mentioned above.

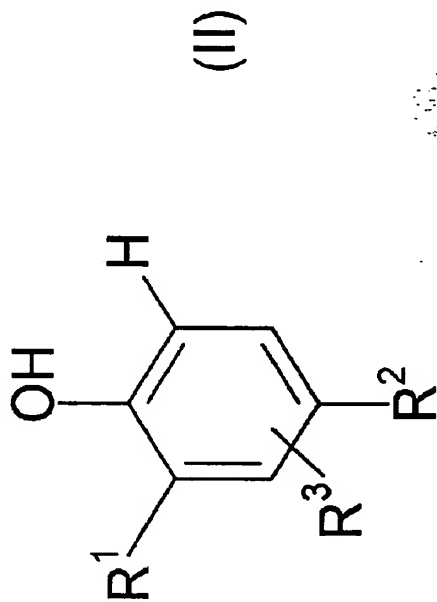
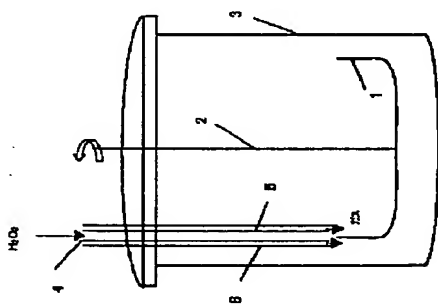
The hydrogen peroxide is added in the presence of a carboxylate salt of formula (III).

[R<sup>4</sup>-C(=O)-O-]<sub>n</sub>Mn<sup>+</sup> (III);

R<sup>4</sup> = phenyl substituted by cycloalkyl, unsubstituted phenyl, alkyl substituted by alkyl which is substituted by unsubstituted alkyl and phenyl, unsubstituted alkenyl, unsubstituted cycloalkyl, or

2004-046543/05	<p>alkyl; M = alkali-metal atom or alkaline-earth metal atom; and n = 1 when M is alkali-metal atom, and 2 when M is alkaline-earth atom.</p> <p>Preferred Conditions: The hydrogen peroxide is added in the presence of a base, using an addition pipe positioned at the bottom of the liquid level of the aqueous medium. The addition pipe is a double pipe (4) having inner and outer pipes (5,6). An inert gas is blown into the outer pipe.</p>	
----------------	---	--

JP 2003171325-A+2



(6pp3007DwgNo.1/1)